

# PATENT SPECIFICATION

DRAWINGS ATTACHED

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Int. Cl.: —C 23 c 3/02//H 01 f

## COMPLETE SPECIFICATION

### Magnetic Recording Medium and Method of Manufacture

We, CLEVITE CORPORATION, a corporation organized and existing under the laws of the State of Ohio of 17000 St. Clair Avenue, Cleveland, Ohio, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates generally to a magnetic recording medium, more particularly a magnetic recording tape, and to a method of manufacturing the same.

According to the invention there is provided a method of making a magnetic recording medium which comprises the steps of hydrolysing a surface of a polyethylene terephthalate substrate by treating said surface with an aqueous solution consisting essentially of an hydroxide of an alkali metal or of an alkaline-earth metal and a monohydric or polyhydric alcohol soluble and stable in the solution, depositing palladium metal nuclei on the hydrolyzed surface and contacting said surface with an electroless plating solution containing cobalt ions to form on said surface a continuous magnetic layer containing cobalt.

Also according to the invention there is provided a magnetic recording medium comprising a flexible substrate consisting of a hydrolysed polyethylene terephthalate film having a face thereof hydrolyzed by treatment with an aqueous solution consisting essentially of an hydroxide of an alkali metal or of an alkaline-earth metal and a monohydric or polyhydric alcohol and, overlying and firmly bonded to said face a continuous magnetic layer composed of a cobalt-phosphorus alloy including at least 97 per cent

by weight of cobalt and having at the interface between the substrate and the magnetic layer substantially uniformly distributed particles of metallic palladium.

The various features and advantages of the invention will be readily apparent from the following description of exemplary embodiments thereof taken in conjunction with the accompanying drawings in which:

Figure 1 is a transverse sectional view through a segment of a magnetic recording medium according to the present invention diagrammatically illustrated on an enlarged scale;

Figure 2 is a gross enlargement of a small portion of Figure 1;

Figure 3 is a flow diagram of a preferred embodiment of the process of the present invention for the production of a magnetic recording medium as shown in Figures 1 and 2; and

Figure 4 is a flow diagram similar to Figure 3 and showing a modified embodiment of the process of this invention.

Referring now to the drawings and first particularly, to Figures 1 and 2, there is here shown diagrammatically in transverse section a metal-clad plastic recording tape in accordance with the present invention having a polyester base or substrate and a magnetic layer of cobalt-base alloy coating adherently disposed on a major surface thereof.

As shown in Figure 2, there are a plurality of growth nuclei, specifically minute particles of metallic palladium (grossly exaggerated in size to permit illustration), adhered to the plastic base at the interface between the base and the magnetic layer. For best results, these growth nuclei must

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be present in a certain minimum density on the surface of the plastic base and the spacing between nuclei must not exceed a certain limiting value in order to facilitate the deposition of a magnetic layer which is substantially free of discontinuities and which firmly adheres to the plastic base. Both of these latter characteristics are essential to the practical utility of the finished article as an information storage medium.

The manner in which the palladium growth nuclei are provided on the surface of the plastic substrate is described in detail hereinbelow. Optimum results have been obtained where there are a minimum of 13 such nuclei, having a transverse dimension of 50 Angstrom units or more, on each square micron of surface area of the plastic base or substrate, and where the average spacing between individual nuclei does not exceed 2800 Angstrom units.

As previously mentioned, the substrate material employed as the base for the magnetic layer is a linear saturated polyester film, for example, a poly (alkylene) terephthalate and, specifically poly (ethylene) terephthalate.

Linear saturated polyesters of the type satisfactory for use in the present invention are well-known in the art; for details of the production of such polymeric materials reference may be had to U.S. Patents Nos. 2,465,319; 2,850,483; and 2,857,363.

The presently preferred material for use as the substrate is polyethylene terephthalate which may be conveniently produced by an ester interchange reaction between methyl terephthalate and ethylene glycol in the presence of a catalyst, such as metallic sodium.

Suitable polyester films are commercially available under a variety of trade names including the Trade Mark "Mylar" (DuPont), "T-16" (Eastman Kodak), and the Trade Mark "Melinex" (Imperial Chemical Industries Limited). While these films are supplied in a wide assortment of dimensions for use as the substrate in a magnetic recording tape, the material preferably is on the order of .5 to 10 mils in thickness and from about 1/8 inch to about 48 inches in width, and has an indeterminate length, depending upon convenience or necessity, adapting the tape for storage on a reel or similar device.

The preferred cobalt-base magnetic layer contains no less than about 97 percent cobalt and the balance phosphorus. Small quantities of nickel and/or iron may be included but have the effect of lowering the coercivity of the magnetic layer. When applied in the manner contemplated by the present invention and described hereinbelow, the cobalt-phosphorus alloy magnetic layer has a unique and highly advantageous combination of magnetic characteristics, namely, as previously mentioned, high permeability, high remanence, and high coercivity. Of equal or

greater importance from the standpoint of information storage capacity and sharpness of definition, the magnetic layer exhibits a substantially square magnetic hysteresis loop.

To produce the unique product described hereinabove in conjunction with Figures 1 and 2, the process of this invention combines three principal phases:

- (1) a particular surface pre-treatment of the substrate;
- (2) the application of catalytic growth nuclei to the pre-treated surface of the substrate; and,
- (3) deposition on that substrate surface of a magnetic layer by the auto-catalytic reduction from an electroless plating solution containing complexed cobalt ions, and preferably also containing hypophosphite ions which serve both as a reducing agent and a source of phosphorus for the magnetic layer.

The details of a preferred embodiment of the method of this invention will now be described with continuing reference to the flow diagram constituting Figure 3 of the drawings.

#### Hydrolysis

In the illustrated embodiment one mil thick "Mylar" tape one inch wide was employed as the substrate. A 100-foot reel of this tape was subjected to a particular pre-treatment step adapted to achieve substantially uniform hydrolysis of the tape surface. This was accomplished by immersion of the tape in a hot aqueous caustic solution composed of 200 grams per liter of sodium hydroxide and 150 milliliters per liter of propylene glycol. With this particular solution, at a temperature of 80 to 85° C., an immersion period of three minutes provided adequate hydrolysis. It will be appreciated, however, that the constituency of the solution, its concentration and temperature, as well as the particular identity of the substrate film, are variable, inter-related parameters all influencing the required time of immersion.

In the hydrolyzing solution, any alkali or alkaline earth metal hydroxide can be used in place of sodium hydroxide, and in lieu of propylene glycol any monohydric or polyhydric alcohol soluble and stable in the solution at its operating temperature might be employed. Examples of suitable alcohols are ethylene glycol, diethylene glycol, and ethyl alcohol. In this connection, however, it should be pointed out that, in addition to limitations imposed by solubility and stability, the more volatile alcohols, such as ethyl alcohol, create a problem due to their tendency to evaporate in use.

#### Sensitization

Following hydrolysis, the tape was rinsed

in water to remove any residual caustic solution and then sensitized by immersion in an acid solution of stannous chloride ( $\text{SnCl}_2$ ). In the exemplary embodiment the solution consisted of 70 grams per liter of stannous chloride, 50 milliliters per liter of 36 per cent concentrated hydrochloric acid ( $\text{HCl}$ ) and 0.5 gram per liter of sodium lauryl sulfate; immersion of the tape in the solution was for two minutes at room temperature although, here again, the time is subject to variation.

The presence of stannous ion ( $\text{Sn}^{++}$ ) is indispensable to operativeness of the solution to sensitize the tape surface for subsequent adsorption of catalytic nuclei as hereinafter described; however, it has been found that although stannic ions ( $\text{Sn}^{+++}$ ) are ineffective in themselves to sensitize the tape surface, their presence in the solution increases the adsorption of tin and, as an ultimate result, increases the adsorption of catalytic nuclei deposited in a subsequent step. As little as one gram per liter of stannic chloride is effective to produce some increase in the tin adsorption. Moreover, in place of the stannous and stannic chlorides, other halide salts of tin may be employed.

#### Activation

Following sensitization, the tape was rinsed in water and immersed in an activating bath made up of an aqueous solution of 10 grams per liter of silver nitrate. Immersion of the tape in the activating bath was continued for a period of about one minute, although this might be varied from 15 seconds to 5 minutes. During this time the reducing action of stannous ions adsorbed on the surface of the tape causes the deposition of isolated particles of silver-silver oxide material which adhere strongly to the surface.

#### Seeding

Once again the tape was rinsed in water and then seeded with catalytic growth nuclei by immersion in an acidic solution of palladium chloride. Here the silver particles present on the surface of the tape as a result of the activation step are replaced by palladium particles, producing the metallic palladium nuclei hereinbefore mentioned and described.

The seeding solution in the illustrated embodiment consisted of 0.1 gram per liter of palladium chloride and 1.0 milliliter per liter of 36 percent concentrated hydrochloric acid. Immersion of the tape in the palladium chloride solution was for one minute, but may be varied from 15 seconds to 5 minutes.

While palladium chloride is the preferred catalyst, any palladium halide salt may be used.

Following seeding, the tape was rinsed in

water to remove the excess solution; the rinse did not disturb the growth nuclei which were strongly adsorbed on the tape surface.

In the specific example under discussion, complete coverage and satisfactory subsequent deposition of the cobalt-phosphorus magnetic layer were obtained when there were at least 13 palladium nuclei, larger than 50 Angstrom units, per square micron of substrate surface and with a maximum distance of 2700 Angstrom units between adjacent palladium nuclei after the seeding step in the process. The number and spacing of the nuclei were verified by electron micrographs (36,500 X) of samples of the substrate surface following the seeding operation.

During the preliminary or pre-plating steps thus far described it is essential that there be no physical contact with the particular surface of the tape on which the magnetic layer is to be applied. To this end the tape was passed through the various solutions while supported on suitably disposed and arranged guide rolls, the tape being given a 180° twist between rollers involving a change in direction of travel.

As indicated previously, the various times of immersion are not in any way critical; however, each should be long enough to insure complete wetting of the tape surface and sufficient time for the particular reaction involved to occur. Where the pre-treatment is carried on as a continuous process involving passage of a continuous tape over guide rolls through the various solutions, the time of immersion, of course, can be regulated by controlling the transport rate of the tape in conjunction with adjustment of the length of run of tape immersed at one time in a particular solution.

#### Plating

Having thus prepared the tape, the cobalt-phosphorus alloy magnetic layer was then deposited on the surface thereof by electroless plating in the following manner.

Generally stated, plating of the cobalt-phosphorus alloy is accomplished by passing the prepared tape through an electroless plating solution containing complexed cobalt ions and a hypophosphite reducing agent. In the described embodiment plating solutions were used which had the following range of composition:

cobalt sulfate . $7\text{H}_2\text{O}$	28—32 gms./l.
ammonium chloride	50—100 gms./l.
sodium potassium tartrate	50—75 gms./l.
sodium hypophosphite	20—25 gms./l.

The pH of plating solutions of this composition was adjusted to between 8.7 and 8.9 with ammonium hydroxide. The alloy plated from solutions having concentrations with the ranges set forth above contained

from 1 to 1.5 weight percent phosphorus and the balance cobalt.

An alternative plating bath composition known to be satisfactory is as follows:

- |   |   |                |
|---|---|----------------|
| 5 | cobalt sulfate . 7H <sub>2</sub> O      | 30 gms./l.     |
|   | ammonium chloride                       | 50 gms./l.     |
|   | sodium potassium tartrate               | 50 gms./l.     |
|   | sodium hypophosphite . H <sub>2</sub> O | 20 gms./l.     |
|   | ammonium hydroxide                      | to give pH 10. |

- 10 Operating at a temperature of 68—70° C. this preferred plating solution yielded a uniform continuous plate in the order of 20 micro-inches in thickness in a plating time of about 12 minutes.

- 15 While the chemical aspects of the plating step are important to the formation of a satisfactory magnetic coating, of equal and perhaps greater importance are the physical conditions under which plating is carried out. It is essential that the tape be brought into contact with the plating solution without subjecting it to any physical stresses such as would stretch, twist, or otherwise physically distort the tape except for simple large radius bending. The reason for this will be apparent in the light of an explanation of what is believed to be the manner in which the plating forms and is bonded to the substrate.

- 20 While the tape is in contact with the plating solution, the palladium nuclei catalyze the reduction of metallic cobalt, alloyed with phosphorus from the sodium hypophosphite reducing agent, and serve as nucleation centers for the formation of an isotropic layer of the alloy which conforms precisely to the microscopic topographical features of the tape surface. At this stage the adherence between the magnetic layer and the substrate is rather tenuous; it is believed to be established by the Vander Walls forces which are effective because of the very close approach of the layer and substrate. Consequently, any distortion of the substrate or other effect which tends to cause relative displacement between the plated layer and substrate before the plated layer is permanently bonded in the manner hereinafter described precludes the formation of a proper bond between the substrate and the plated layer. Therefore, the plated layer and the tape should be maintained against relative displacement until permanent bonding is achieved.

- 25 In the continuous plating of a tape of indeterminate length alluded to hereinabove, the physical conditions pre-requisite to satisfactory electroless plating were fulfilled by using a four-inch-diameter U-tube of a borosilicate glass, a stock industrial item, as the plating vessel. To provide a submerged run of tape three feet in length for the particular described embodiment, the legs of the U-Tube were increased in length by joining straight glass tubes thereto. To maintain the tape against any distortion except simple large radius bending while in the plat-

ing solution, a flat, two inch wide strip of polytetrafluoroethylene curved to the inner radius of the U-Tube was inserted into the bend of the U-Tube. The tape travelled through the electroless plating solution in the U-Tube with the not-to-be-plated surface riding firmly against the Teflon U-bend, passing over synchronously driven feed and take-up reels to preclude stretching of the tape.

A standard heating mantle enveloped the U-Tube to enable temperature control of the solution and conventional means were utilized for continuous filtration of the electroless plating bath.

Bonding of the cobalt-phosphorus layer to the substrate may be accomplished by natural aging, which may vary from a few minutes to a day at room temperatures or, preferably, by heat treatment of the tape to accelerate the aging process. Heat treatment involves heating the tape to a temperature of about 70° C. for from 1 to 60 minutes immediately after plating and prior to rinsing. After a final water rinse the tape is then dried and stored for use.

The sensitization and activation steps of the method may be eliminated from the method described by modification of the seeding step. A flow diagram for this modified embodiment of the invention is illustrated in Figure 4, from which it will be seen that the hydrolysis and the plating steps are the same as illustrated in Figure 3. In this variant, seeding is accomplished by immersing the hydrolyzed tape, after rinsing, in a catalytic metal sol. The tape is then rinsed in water, plated, and finished in the same manner as in the Figure 3 method.

For additional information as to the use of catalytic metal sols for seeding substrates prior to electroless plating reference may be had to U.S. Patent No. 3,011,920.

A specific example of seeding of "Mylar" tape with palladium growth nuclei deposited from a sol is as follows.

A palladium sol was prepared by reduction of palladium chloride solution (pH adjusted to 5) with Formalin at 50° C. The hydrolyzed tape was immersed in the freshly-prepared sol for 1 to 2 minutes, rinsed with water and then plated in an electroless cobalt bath, as described above.

In this modified embodiment of the present process, the number of catalytic nuclei per unit area and spacing therebetween are maintained as described in connection with the Figure 3 embodiment.

Cobalt-phosphorus alloy layers deposited in accordance with the present invention are extremely thin, ranging from 5 to 100 micro-inches in thickness depending on various process parameters including the exact bath composition, temperature, time of immersion, and the like. The plating is remarkably uniform

in thickness, fine-grained, and substantially free of discontinuities greater than 1.0 mil in the longitudinal direction of the tape and 0.25 mil in the transverse direction.

5 The following table shows the parameters of several tapes according to the present invention which were produced in accordance with the embodiment of the present process shown in Figure 3 and described in detail

10	Thickness of Magnetic Layer (in micro-inches)		Retentivity or Remanence Flux Density $B_r$ (in Gauss)	
	Coercivity $H_c$ (in Oersteds)			
15	18	410	6,900	
	22	310	4,080	
	24	350	5,960	
	18	290	5,820	
20	22	360	6,000	
	19	420	5,300	
	19	400	6,030	
	18.5	420	5,860	
25	18	450	5,600	
	19	360	4,700	
	19	400	6,540	
	19	460	5,930	
	9	420	11,500	

From the foregoing table it will be evident that each of these tapes had a coercivity of at least substantially 300 Oersteds (290 being the lowest) and a retentivity, or remanent flux density, higher than 4,000 Gauss. Due to the higher coercivity and retentivity, the tapes of the present invention are capable of higher output signals (with a higher signal-to-noise ratio) on playback and higher "packing" densities of recorded binary digits, as well as being adapted for slower speed playback operation. It has been determined that the tape of the present invention is capable of recording with much smaller pulse widths—1/4 to 1/3 of the pulse width obtainable with conventional magnetic record media. As a result of the narrow pulse capability, the present tape can record up to three times the amount of information per unit of tape length than conventional tapes. Specifically, conventional magnetic tapes are capable of storing 250 to 500 binary digits or "bits" per inch as compared to 1,000 to 1,500 or more bits per inch for magnetic tapes in accordance with the present invention.

These improved characteristics make the tape of the present invention especially advantageous for instrumentation applications, such as in computers.

While there have been described what at present are believed to be the preferred embodiments of this invention, it will be obvious to those skilled in the art that various changes and modifications may be made therein without departing from the invention, and it is aimed, therefore, to cover in the appended claims all such changes and modi-

fications as fall within the scope of the invention.

#### WHAT WE CLAIM IS:—

1. A method of making a magnetic recording medium which comprises the steps of hydrolyzing a surface of a polyethylene terephthalate substrate by treating said surface with an aqueous solution consisting essentially of an hydroxide of an alkali metal or of an alkaline-earth metal and a monohydrate or polyhydric alcohol soluble and stable in the solution, depositing palladium metal nuclei on the hydrolyzed surface and contacting said surface with an electroless plating solution containing cobalt ions to form on said surface a continuous magnetic layer containing cobalt.

2. A method as claimed in claim 1 wherein said plating solution contains complexed cobalt ions and a hypophosphite reducing agent whereby said magnetic layer comprises an alloy of cobalt and phosphorus.

3. A method as claimed in claim 2 wherein said alloy comprises not less than 97 percent by weight of cobalt.

4. A method as claimed in any preceding claim wherein the average number of said nuclei of a size larger than 50 Angstrom units deposited per square micron of said surface is at least 13 and the maximum distance between adjacent nuclei is less than 2800 Angstrom Units.

5. A method as claimed in any preceding claim, including the step of maintaining said magnetic layer and said surface against relative displacement during the formation of the magnetic layer and for a period of time thereafter until the layer is firmly bonded to said surface.

6. A method as claimed in claim 5 wherein bonding of said magnetic layer to said surface is accelerated by the application of heat while the magnetic layer and surface are maintained against relative displacement.

7. A method as claimed in any of the preceding claims wherein said nuclei are deposited by immersing the substrate in a palladium sol.

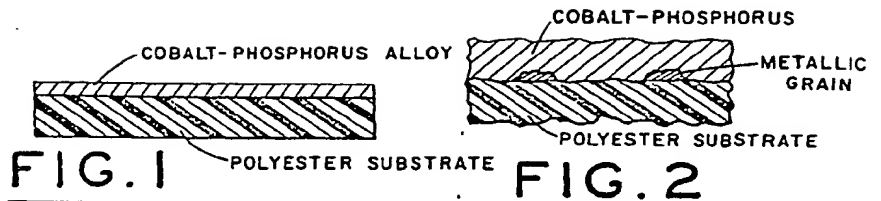
8. A method as claimed in any of claims 1 to 6 wherein said nuclei are deposited by immersing the substrate in sequence in an aqueous acid solution of a stannous halide, in a silver salt in aqueous solution, and in an aqueous acid solution of a palladium halide.

9. A method of making a magnetic recording tape which comprises the steps of: contacting at least one major surface of a polyethylene terephthalate film with a hot caustic solution containing an alkylene glycol until said surface is hydrolyzed; chemically depositing on said hydrolyzed surface uniformly distributed catalytic nuclei of metallic palladium; thereafter contacting said surface

- with an electroless plating solution containing complexed cobalt ions and a hypophosphite reducing agent to form a continuous magnetic layer of metallic cobalt-phosphorus alloy contiguous with and precisely conforming to the microscopic topographical features of said surface while maintaining said layer and said surface against relative displacement; and, while the magnetic layer and said surface are maintained against relative displacement, heating the film and the magnetic layer to a temperature of about 70°C. for from 1 to 60 minutes.
10. A method as claimed in claim 9, wherein the average number of palladium nuclei of a size larger than 50 Angstrom units deposited per square micron of said surface is at least 13, and the maximum distance between adjacent palladium nuclei is less than 2800 Angstrom units.
11. A method as claimed in claim 9 or 10 wherein said caustic solution consists of propylene glycol and an alkali, or alkaline earth, hydroxide.
12. A method as claimed in claim 9, 10 or 11 wherein said caustic solution consists of
- |                  |            |
|------------------|------------|
| sodium hydroxide | 200 gm./l. |
| propylene glycol | 150 ml./l. |
13. A method as claimed in claim 9, 10, 11 or 12 wherein said palladium metal nuclei are deposited by: contacting said surface of the substrate with a sensitizing solution consisting of:
- |                       |             |
|-----------------------|-------------|
| stannous chloride     | 70 gms./l.  |
| 36% Conc. HCl         | 50 ml./l.   |
| sodium lauryl sulfate | 0.5 gms./l. |
- subsequently contacting said surface with an activating solution consisting of:
- |                |            |
|----------------|------------|
| silver nitrate | 10 gms./l. |
|----------------|------------|
- and subsequently contacting said surface with a seeding solution consisting of:
- |                    |            |
|--------------------|------------|
| palladium chloride | 0.1 gm./l. |
| 36% Conc. HCl      | 1.0 ml./l. |
14. A method as claimed in any one of claims 8 to 12 wherein said electroless plating solution consists essentially of:
- |  |            |  |                |
|--|------------|--|----------------|
| cobalt sulfate ( $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ ) | 30 gms./l. | ammonium chloride                            | 50 gms./l.     |
|  |            | potassium sodium tartrate                    | 50 gms./l.     |
|  |            | sodium hypophosphite                         |                |
|  |            | ( $\text{NaPO}_3 \cdot \text{H}_2\text{O}$ ) | 20 gms./l.     |
|  |            | ammonium hydroxide                           | to give pH 10. |
15. A method of making a magnetic recording medium substantially as herein described.
16. A magnetic recording medium when made by the method claimed in any one of the preceding claims.
17. A magnetic recording medium comprising a flexible substrate consisting of a polyethylene terephthalate film having a face thereof hydrolyzed by treatment with an aqueous solution consisting essentially of an hydroxide of an alkali metal or of an alkaline-earth metal and a monohydric or polyhydric alcohol and, overlying and firmly bonded to said face a continuous magnetic layer composed of a cobalt-phosphorus alloy including at least 97 percent by weight of cobalt and having at the interface between the substrate and the magnetic layer substantially uniformly distributed particles of metallic palladium.
18. A medium as claimed in claim 17 wherein there are at least 13 of said particles larger than 50 Angstrom units per square micron of interface area, the maximum distance between adjacent particles being less than 2800 Angstrom units.
19. A medium as claimed in claim 14 or claim 15 wherein said magnetic layer has a coercivity of at least 300 Oersteds and a retentivity of at least 4,000 Gauss.
20. A medium as claimed in any one of claims 17, 18 or 19 wherein said magnetic layer has a thickness not greater than 100 micro-inches.
21. A magnetic recording medium substantially as hereinbefore described.
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303/306 High Holborn, London, W.C.1.

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HYDROLYSIS: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
 200 g/l NaOH  
 150 ml/l propylene glycol  
 AT 80-85°C FOR 3 MINUTES

SENSITIZATION: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
 7G g/l SnCl<sub>2</sub>  
 50 ml/l HCl  
 5 gm/l Na-lauryl-SO<sub>4</sub>  
 AT ROOM TEMPERATURE FOR 2 MINUTES

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ACTIVATION: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
 0.1 g/l AgNO<sub>3</sub>

SEEDING: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
 0.1 g/l PdCl<sub>2</sub>  
 1.0 ml/l HCl  
 AT ROOM TEMPERATURE FOR 2 MINUTES

PLATING: IMMERSE MYLAR SUBSTRATE IN SOLUTION:

CoSO <sub>4</sub> 7H <sub>2</sub> O	30 g/l
NaK-TARTRATE	50 g/l
NH <sub>4</sub> Cl	50 g/l
NaH <sub>2</sub> PO <sub>4</sub> H <sub>2</sub> O	20 g/l
NH <sub>4</sub> OH	TO adjust pH

AT 65-70°C AND pH 8-11 FOR 10 TO 15 MINUTES  
 TO PLATE 20-25 MICRO-INCHES

BONDING:

AGE  
 OR  
 HEAT TREAT AT 70°C FOR 1 TO 60 MINUTES

FIG. 3

1045086 COMPLETE SPECIFICATION

2 SHEETS *This drawing is a reproduction of  
the Original on a reduced scale  
Sheets 1 & 2*

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HYDROLYSIS: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
200g/l NaOH  
150ml/l propylene glycol  
AT 80-85°C FOR 3 MINUTES

SEEDING: IMMERSE MYLAR SUBSTRATE IN  
PALLADIUM SOL FOR  
1 TO 2 MINUTES

PLATING: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
CoSO<sub>4</sub> 7H<sub>2</sub>O 30 g/l  
NaK-TARTRATE 50 g/l  
NH<sub>4</sub>Cl 50 g/l  
NaH<sub>2</sub>PO<sub>4</sub> H<sub>2</sub>O 20 g/l  
NH<sub>4</sub>OH TO adjust pH  
AT 65-70°C AND pH 8-11 FOR 10 TO 15 MINUTES  
TO PLATE 20-25 MICRO-INCHES

BONDING:  
AGE  
OR  
HEAT TREAT AT 70°C FOR 1 TO 60 MINUTES

FIG. 4

ES

ES



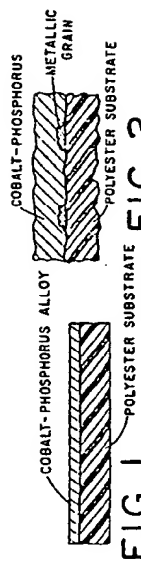


FIG. 1

HYDROLYSIS: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
200 g/l NaOH  
150 ml/l propylene glycol  
AT 80-85°C FOR 3 MINUTES

SENSITIZATION: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
70 g/l SnCl<sub>2</sub>  
50 ml/l HCl  
5 gm/l Na-lauryl-SO<sub>4</sub>  
AT ROOM TEMPERATURE FOR 2 MINUTES  
30 JUL 1963

ACTIVATION: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
0.1 g/l AgNO<sub>3</sub>

SEEDING: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
0.1 g/l PdCl<sub>2</sub>  
1.0 ml/l HCl  
AT ROOM TEMPERATURE FOR 2 MINUTES

PLATING: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
CoSO<sub>4</sub> 7 H<sub>2</sub>O 30 g/l  
NaK-TARTRATE 50 g/l  
NH<sub>4</sub>Cl 50 g/l  
NaH<sub>2</sub>PO<sub>4</sub> H<sub>2</sub>O 20 g/l  
NH<sub>4</sub>OH TO adjust pH  
AT 65-70°C AND pH 8-11 FOR 10 TO 15 MINUTES  
TO PLATE 20-25 MICRO-INCHES

BONDING:  
AGE  
OR  
HEAT TREAT AT 70°C FOR 1 TO 60 MINUTES

FIG. 3

HYDROLYSIS: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
200 g/l NaOH  
150 ml/l propylene glycol  
AT 80-85°C FOR 3 MINUTES

SEEDING: IMMERSE MYLAR SUBSTRATE IN  
PALLADIUM SOL FOR  
1 TO 2 MINUTES

PLATING: IMMERSE MYLAR SUBSTRATE IN SOLUTION:  
CoSO<sub>4</sub> 7 H<sub>2</sub>O 30 g/l  
NaK-TARTRATE 50 g/l  
NH<sub>4</sub>Cl 50 g/l  
NaH<sub>2</sub>PO<sub>4</sub> H<sub>2</sub>O 20 g/l  
NH<sub>4</sub>OH TO adjust pH  
AT 65-70°C AND pH 8-11 FOR 10 TO 15 MINUTES  
TO PLATE 20-25 MICRO-INCHES

BONDING:  
AGE  
OR  
HEAT TREAT AT 70°C FOR 1 TO 60 MINUTES

FIG. 4

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